

# PATENT ABSTRACTS OF JAPAN

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## (54) MOISTURE-CURABLE RESIN COMPOSITION

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a moisture-curable resin composition of which the surface tackiness disappears in a short time as compared with the existing moisture-curable resin composition.

**SOLUTION:** This moisture-curable resin composition is composed of 100 pts.wt. of an organic polymer (a) containing a silyl group and having at its molecular terminal or on its side chain at least one functional group having a silicon atom bound to a hydrolyzable group per molecule and 0.1-20 pts.wt. of a tin compound (b) of a carboxylic acid which is expressed in general formula (1) below (wherein, R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup> are each a 1-10C hydrocarbon group that may be identical or different) where the α carbon atom of the carboxylic acid is a tertiary carbon.



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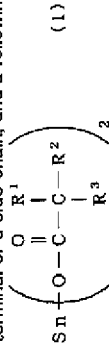
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## CLAIMS

[Claim(s)]

[Claim 1]Silyl group content organicity polymer (a) 100 weight section which has one basis which has the silicon atom combined with a hydrolytic basis even if small [ per molecule ] to a molecular terminal or a side chain, and a following general formula (1) : [Formula 1]



(Among a formula, R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup> are hydrocarbon groups with 1-10 carbon atoms, respectively, and) differing, even if R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup> are the same to mutual --- \*\*\* --- a moisture curing type resin composition, wherein the carbon atom of the alpha position of carboxylic acid expressed consists of the tin compound (b) 0.1 of the carboxylic acid which is the third class carbon - 20 weight sections. [Claim 2]The moisture curing type resin composition according to claim 1 which is a moisture curing type resin composition of 2 liquid type which consists of a main agent component (A) containing said silyl group content organicity polymer (a), and a curing catalyst ingredient (B) containing said tin compound (b) ingredient.

[Claim 3]The moisture curing type resin composition according to claim 1 or 2 in which said silyl group content organicity polymer (a) is what uses a polymer of polyether and an ethylenic unsaturated compound, or a polymer of diene series as a main chain.

[Claim 4]The moisture curing type resin composition according to any one of claims 1 to 3 in which said tin compound (b) is SUTANASU screw pivalate or SUTANASU screw neo decanoate.

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] Especially this invention relates to a moisture curing type resin composition suitable 2 liquid type to use as a sealing agent, adhesives, etc. with which it hardens about a moisture curing type resin composition for a short time after mixing a main agent component and a curing catalyst ingredient, and surface tackiness disappears for a short time.

[0002]

[Description of the Prior Art] As a useful hardening resin constituent, the thing using the organic polymer which has a hydrolytic silicon content group which uses polyether, polyester or an ethylenic unsaturated compound, the polymer of diene series, etc. as a main chain, and for which a bridge can be constructed as the main ingredients is known by adhesives and the sealing agent. These are stiffened by forming a siloxane bond. The hardening resin constituent using the organic polymer which has this hydrolytic silicon content group is excellent in hardenability, storage stability, weatherability, etc. As a curing catalyst of the organic polymer which has said hydrolytic silicon content group, The organic metallic compound of others, such as organic tin (IV) compounds, such as carboxylic acid tin (II) compounds, such as 2-ethylhexanoic acid tin and n-tin octylate, dibutyltin dilaurate, and dibutyltin maleate, naphthenic acid iron, and lead octylate, is used. Especially, since it excels in a cure rate and hardened material nature, the organic tin (IV) compound and the carboxylic acid tin (II) compound are used widely.

[0003]

[Problem(s) to be Solved by the Invention] However, since the influence which does each of organic tin compounds and organic lead compounds to environment and a human body is great, when using it, sufficient cautions are needed. moreover — conventionally, carboxylic acid tin (II), such as 2-ethylhexanoic acid tin and n-tin octylate, are mainly used as an organic tin compound — \*\*\*\* (JP.S61-60867.B). The carbon atom of the alpha position of carboxylic acid was carboxylic acid which is first-class carbon or the second class carbon, and all were deactivated with the humidity in the air, etc., and had the problem that surface tackiness was not canceled for a long time. Therefore, development of the curing catalyst which has a practical cure rate and by which surface tackiness is canceled for a short time was desired.

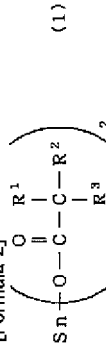
[0004]

[Means for Solving the Problem] Then, wholeheartedly, as a result of research, this invention persons find out a moisture curing type resin composition which has the fast curability which is equal to 2-ethylhexanoic acid tin currently conventionally used as a curing catalyst, n-tin octylate, etc., and surface tackiness cancels for a short time, and came to complete this invention.

[0005] That is, this invention provides the following moisture curing type resin composition.

(1) Silyl group content organicity polymer (a) 100 weight section which has one basis which has the silicon atom combined with a hydrolytic basis even if small [per molecule] to a molecular terminal or a side chain, and following general formula (1): [0006]

[Formula 2]



[0007] (Among a formula, R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup> are hydrocarbon groups with 1-10 carbon atoms,

respectively, and) differing, even if R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup> are the same to mutual — \*\*\*\* — a moisture curing type resin composition, wherein the carbon atom of the alpha position of carboxylic acid expressed consists of the tin compound (b) 0.1 of the carboxylic acid which is the third class carbon — 20 weight sections.

(2) A moisture curing type resin composition given in (1) paragraph which is a moisture curing type resin composition of 2 liquid type which consists of a main agent component (A) containing said silyl group content organicity polymer (a), and a curing catalyst ingredient (B) containing said tin compound (b) ingredient.

(3) A moisture curing type resin composition given in (1) or (2) paragraphs in which said silyl group content organicity polymer (a) is what uses the polymer of polyether and an ethylenic unsaturated compound, or the polymer of diene series as a main chain.

(4) A moisture curing type resin composition given in either of the (1) - (3) paragraphs in which said tin compounds (b) are the SUTANASU screw pivalate or SUTANASU screw neo decanoate.

[0008]

[Embodiment of the Invention] The basis which has the silicon atom which combined with the hydrolytic basis the organic polymer (a) used for this invention. Even if small [to a molecular terminal or a side chain / per molecule] in (it may be hereafter called the silicon group combined with the hydrolytic basis), it is an organic polymer which it has one piece, and an alkylene oxide polymer thru/or polyether, an ether ester block copolymer, etc. are mentioned as a main chain of this polymer. The polymer of an ethylenic unsaturated compound and diene series, etc. are mentioned. The liquefied thing of these main chain polymer is preferred at a room temperature.

[0009] As said alkylene oxide polymer thru/or polyether, [CH<sub>2</sub>CH<sub>2</sub>O] n [CH(CH<sub>3</sub>) CH<sub>2</sub>O] n [CH(CH<sub>2</sub>H<sub>3</sub>) CH<sub>2</sub>O] n [CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O] What has one sort of repeating units, such as n, or two sorts or more is illustrated. Here, n is two or more integers. These alkylene oxide polymers thru/or polyether may be used independently, and may use two or more sorts together.

[0010] As a polymer of an ethylenic unsaturated compound and diene series. Homopolymers, such as ethylene, propylene, acrylic ester, methacrylic acid ester, vinyl acetate, acrylonitrile, styrene, isobutylene, butadiene, isoprene, and chloroprene, or these two or more sorts of copolymers are mentioned. More specifically Polybutadiene, a styrene butadiene copolymer, an acrylonitrile butadiene copolymer, An ethylene-butadiene copolymer, ethylene propylene rubber, an ethylene-vinylacetate copolymer, An ethylene-(meta) acrylic ester copolymer, polyisoprene, A styrene isoprene copolymer, an isobutylene-isoprene copolymer, polychloroprene, a styrene chloroprene copolymer, an acrylonitrile chloroprene copolymer, polyisobutylene, polyacrylic ester, polymethacrylic acid ester, etc. are mentioned. These may be used independently or may use two or more kinds together.

[0011] The silicon group combined with said hydrolytic basis is a basis which causes a condensation reaction by using a catalyst etc. if needed under existence of humidity and a cross linking agent. Specifically, a halogenation silyl group, alkoxy silyl groups, an alkenyl oxyisilyl group, an acyloxy silyl group, an amino silyl group, an amino oxyisilyl group, an oxime silyl group, and an amide silyl group are mentioned. Here, the number of these hydrolytic bases combined with one silicon atom is chosen from the range of 1-3. The number of the hydrolytic bases combined with one silicon atom may be one, and that of them may be [two or more]. Furthermore, the hydrolytic basis and the non-hydrolytic basis may combine with one silicon atom. As a silicon group combined with the hydrolytic basis, handling is an easy point, and especially alkoxy silyl groups (mono- alkoxy silyl groups, a dialkoxy silyl group, and the Tori alkoxy silyl groups are included) is preferred. The silicon group combined with the hydrolytic basis may exist in the end of a polymer molecule, or may exist in the side chain. Even if the silicon group combined with the hydrolytic basis has few polymers per molecule, it is preferred that it averages per molecule from a point of a cure rate and hardened material nature, and there are 1.5 or more pieces with one piece although it is good. A method publicly known as a method of combining with said main chain polymer the silicon group combined with the hydrolytic basis is employable.

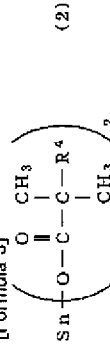
[0012] Although the molecular weight of the organic polymer (a) used by this invention does not have restrictions in particular, the thing of Polymer Division is hyperviscosity, and since it becomes

difficult [ a use top ] when it is considered as a hardening resin constituent, 30000 or less are too desirable [ a thing ] as a number average molecular weight. Although such an organic polymer can be manufactured by a publicly known method, commercial items, such as KANEKA MS polymer by Kaneka Corp., may be used for it.

[0013]One sort of the tin compound (it may be hereafter called carboxylic acid tin salt) of the carboxylic acid whose carbon atom of the alpha position of carboxylic acid expressed with a general formula (1) is the third class carbon as a curing catalyst (b) used for this invention, or two sorts or more are used. In a general formula (1), as a hydrocarbon group with 1-10 carbon atoms shown by R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup>. For example, straight chain shape or branched state alkyl groups, such as methyl, ethyl, propyl, isopropyl, butyl, isobutyl, sec-butyl, pentyl, heptyl, octyl, 2-ethylhexyl, nonyl, and decyl, are mentioned.

[0014]In the carboxylic acid tin salt expressed with a general formula (1), it is a following general formula (2) : [0015]

[Formula 3]



[0016]What is expressed with (inside of formula and R<sup>4</sup> is a hydrocarbon group with 1-10 carbon atoms) is preferred. Here, a carbon atom of an alpha position in carboxylic acid tin salt expressed with a general formula (2) is the third class carbon, and, generally carboxylic acid whose at least two hydrocarbon groups of an alpha position are methyl groups is called neo carboxylic acid. As a hydrocarbon group with 1-10 carbon atoms expressed with R<sup>4</sup>. For example, straight chain shape or branched state alkyl groups, such as methyl, ethyl, propyl, isopropyl, butyl, isobutyl, sec-butyl, pentyl, heptyl, octyl, 2-ethylhexyl, nonyl, and decyl, are mentioned.

[0017]When said carboxylic acid tin salt is illustrated concretely, SUTANASU screw neo decanoate, SUTANASU screw pivalate, SUTANASU screw neo pentanoate, SUTANASU screw neo hexanoate, SUTANASU screw neo heptanoate, SUTANASU screw neo octanoate, SUTANASU screw neo undecanoate, SUTANASU screw neo dodecanoate, SUTANASU screw neo tridecanoate, SUTANASU screw neo tetradecanoate, etc. are mentioned, and these are independent, or can be mixed and used. Such a compound can be manufactured by a publicly known method. In these carboxylic acid tin salt, they are SUTANASU screw pivalate from points, such as the ease of dealing with it on use, and the stability of a compound, and SUTANASU screw neo decanoate. [SUTANASUBISU (2, 2 \*\*JIME chill octanoate)] is preferred.

[0018]As for especially a curing catalyst (b), in a moisture curing type resin composition of this invention, it is preferred to use it at a rate of 0.5 - 10 weight section 0.1 to 20 weight section to silyl group content organicity polymer (a) 100 weight section. Unless quantity of a curing catalyst (b) is less than said range, hardening performance is bad and surface tackiness may not be improved. When said range is exceeded, the physical properties of a hardened material and stability may fail.

[0019]In order to promote hardening in a moisture curing type resin composition of this invention and to improve adhesion to a substrate, Can use publicly known various amino group substitution alkoxy silane compounds or a condensate of those, and specifically, gamma \*\*AMINO propyltrimethoxysilane, gamma \*\*AMINO propyl triethoxysilane, N-(trimethoxysilylpropyl) ethylenediamine, delta-aminobutyl (methyl) diethoxysilane, N, and N' \*\*BISU (trimethoxysilylpropyl) ethylenediamine, these partial hydrolysates, etc. are mentioned. Vinyl alkoxy silane compounds, such as vinyltriethoxysilane, vinyltrimethoxysilane, and a vinyl trisopropoxy silane, can be used for improvement in adhesion to a substrate.

[0020]An additive agent usually added by hardening setup-of-tooling products, such as a bulking agent, colorant, a plasticizer, a hardening accelerator, a concrete retarder, a dripping inhibitor, an antiaging agent, and a solvent, may be further added to a moisture curing type resin composition of this invention.

[0021]As a hardening accelerator, specifically For example, a butylamine, octylamine, Lauryl amine, n-hexadecyl amine, dibutyl amine, monoethanolamine, Diethanolamine, triethanolamine,

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diethylenetriamine, Triethylenetetramine, oleylamine, cyclohexylamine, Benzylamine, diethylamino propylamine, xylene diamine, Triethylenediamine, polyoxy ethylenediamine, polyoxy propylenediamine, Guanidine, diphenylguanidine, 2,4,6-tris(dimethyl aminomethyl) phenol, Amine system organic compounds, such as morpholine, N-methylmorpholine, 2-ethyl-4-methylimidazole, and the 1,8-diazabicyclo (5,4,0) undecene 7 (DBU), are mentioned.

[0022]As a bulking agent, specifically Calcium carbonate, kaolin, talo, Fumed silica, sedimentation nature silica, a silicic acid anhydride, hydrous silicic acids, clay, Calcination clay, glass, bentonite, organic bentonite, a milt balloon, glass fiber, asbestos, a glass filament, grinding quartz, diatomite, aluminum silicate, aluminium hydroxide, a zinc oxide, magnesium oxide, a titanium dioxide, etc. are mentioned.

[0023]Specifically as colorant, iron oxide, carbon black, copper phthalocyanine blue, Phthalocyanine Green, etc. are used.

[0024]As a plasticizer, specifically Dibutyl phthalate, dioctyl phthalate, Phthalic ester, such as butylbenzyl phthalate, dioctyl adipate, Succinic acid dioctyl, succinic acid diisodecyl, diisodecyl sebacate, Ester species of polyol compounds, such as aliphatic-carboxylic-acid ester species, such as butyl oleate, and pentaerythritol ester, Epoxy system plasticizers, such as phosphoric ester, such as trioctyl phosphate and tricresyl phosphate, epoxidized soybean oil, and epoxy stearic acid benzyl, a chlorinated paraffin, etc. are used.

[0025]Specifically as a dripping inhibitor, hydrogenation castor oil, a silicic acid anhydride, organic bentonite, colloidal silica, etc. are used.

[0026]As other additive agents, adhesion grant agents, such as phenol resin and an epoxy resin, an ultraviolet ray absorbent, radical chain inhibitor, a peroxide decomposition agent, various kinds of antiaging agents, etc. are used.

[0027]As for a moisture curing type resin composition of this invention, it is preferred to use it as a moisture curing type resin composition of 2 liquid type which consists of a main agent component (A) containing said silyl group content organicity polymer (a) and a curing catalyst ingredient (B) containing said tin compound (b). In a main agent component (A), besides said silyl group content organicity polymer (a), said amino group substitution alkoxy silane compound, One sort, such as a vinyl alkoxy silane compound, a bulking agent, colorant, a plasticizer, a hardening accelerator, a concrete retarder, a dripping inhibitor, an adhesion grant agent, an ultraviolet ray absorbent, radical chain inhibitor, a peroxide decomposition agent, an antiaging agent, and a solvent, or two sorts or more can be blended suitably. For a curing catalyst ingredient (B), besides said tin compound (b), said amino group substitution alkoxy silane compound, One sort, such as a vinyl alkoxy silane compound, a bulking agent, colorant, a plasticizer, a hardening accelerator, a concrete retarder, a dripping inhibitor, an adhesion grant agent, an ultraviolet ray absorbent, radical chain inhibitor, a peroxide decomposition agent, an antiaging agent, and a solvent, or two sorts or more can be blended suitably. [0028]

[Example]Although working example is given and this invention is explained concretely hereafter, the range of this invention is not limited by this.

[0029]344.5 g (2 mol) of neo decanoic acid (2,2-dimethyloctanoic acid) and the sodium hydroxide solution 170g (sodium hydroxide: 2 mol) are measured in the 1000-ml eggplant flask furnished with an example of manufacture 1 nitrogen introducing pipe. After fully mixing with a magnetic stirrer, added 225 g (1 mol) of stannous chloride, it was made to react at 60 \*\* for 20 minutes, the reaction mixture was extracted with toluene, it condensed under decomposition, and the tin compound a of the fluid of light yellow transparency was obtained.

[0030]This compound was analyzed in FT-IR, and 1738 cm of absorption <sup>-1</sup> of the carbonyl group of neo decanoic acid shifted to 1650 cm <sup>-1</sup>, and checked absorption of tin carbonyl. It checked that it was SUTANASU screw neo decanoate from the result of the next ultimate analysis.

[0031]

	C (%)	H (%)	O (%)	Sn (%)
測定値	52.7	8.2	13.3	25.5
理論値	52.8	8.3	13.1	25.7

[0032]The pivalate 204g (2 mol) and the sodium hydroxide solution 170g (sodium hydroxide: 2 mol) are measured in the 1000-ml eggplant flask furnished with an example of manufacture 2 nitrogen introducing pipe. After fully mixing with a magnetic stirrer, added 225 g (1 mol) of stannous chloride, it

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was made to react at 60 °C for 20 minutes, the reaction mixture was extracted with toluene, it condensed under decompression, and the tin compound b of the fluid of light yellow transparency was obtained.

[0033] This compound was analyzed in FT-IR, and 1738 cm<sup>-1</sup> of the carbonyl group of pivalate shifted to 1650 cm<sup>-1</sup>, and checked absorption of tin carbonyl. It checked that it was the SUTANASU screw pivalate from the result of the next ultimate analysis.

[0034]

	C (%)	H (%)	O (%)	S n (%)
測定値	32.5	5.1	22.0	40.3
理論値	32.8	4.8	21.8	40.5

[0035] To working example 1 - 8 silyl-group content organicity polymer (MS polymer S810 by Kaneka Corp.) 100 weight section, it was shown in Table 1, and number-of-copies (number of weight sections) addition was carried out, the various additive agents shown in Table 1 were kneaded, and the main agent component was prepared. On the other hand, it was shown in Table 1, and number-of-copies (number of weight sections) to silyl group content organicity polymer (MS polymer S810 by Kaneka Corp.) 100 weight section) mixing was carried out, the tin compound a or b and the various additive agents which were obtained in the examples 1 and 2 of manufacture were kneaded, and the curing catalyst ingredient was prepared.

[0036] About the moisture curing type resin composition which mixed the obtained main agent component and the curing catalyst ingredient, a snap time (time until it gels in the mixed second half of both ingredients and mobility is lost), and tack free time (time until the after-mixing surface tackiness of both ingredients is lost) were measured. It went in the thermostatic chamber of the room temperature of 25 °C, and 60% of humidity from preparation of a main agent component and a curing catalyst ingredient, and mixing of both ingredients to hardening. A result is shown in Table 1.

[0037] To the comparative example 1 - 4 silyl-group content organicity polymer (MS polymer S810 by Kaneka Corp.) 100 weight section, it was shown in Table 1, and number-of-copies (number of weight sections) addition was carried out, the various additive agents shown in Table 1 were kneaded, and the main agent component was prepared. On the other hand, it was shown in Table 1, and number-of-copies (number of weight sections) to silyl group content organicity polymer (MS polymer S810 by Kaneka Corp.) 100 weight section) mixing was carried out, 2-ethylhexanoic acid tin or n-tin octylate, and the various additive agents which are used from the former were kneaded, and the curing catalyst ingredient was prepared.

[0038] About the moisture curing type resin composition which mixed the obtained main agent component and the curing catalyst ingredient, a snap time and tack free time were measured like working example 1-8. A result is shown in Table 1.

[0039] The details of the material shown in Table 1 are as follows.

MS polymer S810: silyl group end polyether calcium carbonate: --- bulking agent NOKURAKKU NS-6: --- an antiaging agent (product made from Ouchi Shinko Chemical Industry)  
SUMOIRU P-350: Liquid paraffin (product made from Muramatsu Petroleum)  
Tinvin 327: Ultraviolet ray absorbent (product LFI made from Tokyo Fine chemicals ]-101)  
A-171: Vinyl alkoxy silane compound (made by Nippon Unicar)  
A-1100: Amino group substitution alkoxy silane compound (made by Nippon Unicar)  
lauryl amine: --- Kanto Kagaku extra-pure-reagent bis(2-ethylhexanoic acid)tin: --- the day east --- Transformation --- make --- bis(n-octylic acid)tin: --- the day east --- Transformation --- make [0040] [Table 1]

表 1

組成物成分	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7	実施例 8	比較例 1	比較例 2	比較例 3	比較例 4
MSPリマー-S610	100	100	100	100	100	100	100	100	100	100	100	100
炭酸カルシウム	120	120	120	120	120	120	120	120	120	120	120	120
水素化マン油	6	6	6	6	6	6	6	6	6	6	6	6
ノクソンNS-6	1	1	1	1	1	1	1	1	1	1	1	1
スズミール R-350	15	15	15	15	15	15	15	15	15	15	15	15
チズピン327	1	1	1	1	1	1	1	1	1	1	1	1
A-171	5	5	5	5	5	5	5	5	5	5	5	5
A-1100	5	5	5	5	5	5	5	5	5	5	5	5
酸化チタン	20	20	20	20	20	20	20	20	20	20	20	20
スズミール R-350	5	5	5	5	5	5	5	5	5	5	5	5
ラウリルアミン	1	1	1	1	1	1	1	1	1	1	1	1
酸化合物 <sup>a</sup>	1	1	3	3	5	5	3	2				
酸化合物 <sup>b</sup>												
ビス(2-エチルヘキサン酸)銅												
ビス(4-メチルペンチル酸)銅												
スナバツタタイム(時間)	3.3時間	2.8時間	2.6時間	1.1時間	1.8時間	0.8時間	1.1時間	2.9時間	2.4時間	1.1時間	4.5時間	3.8時間
タックフリータイム(時間)	11時間	7時間	3.0時間	2.6時間	2.3時間	1.7時間	2.2時間	12時間	48時間 以上	48時間 以上	48時間 以上	48時間 以上